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Indian Standard

POTASSIUM PERCHLORATE FOR EXPLOSIVES AND PYROTECHNIC COMPOSITIONS — SPECIFICATION

UDC 662·222·23

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Explosives and Pyrotechnics Sectional Committee, CHD 026

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

Potassium perchlorate is widely used in the manufacture of explosives, pyrotechnic compositions and propellants. It is a strong oxidizing agent. It has a molecular formula $KClO_4$ and molecular mass 138·6.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

POTASSIUM PERCHLORATE FOR EXPLOSIVES AND PYROTECHNIC COMPOSITIONS — SPECIFICATION

1 SCOPE

This standard prescribes requirements and methods of sampling and test for potassium perchlorate for explosives and pyrotechnic industry.

2 REFERENCES

The following Indian Standards are the necessary adjuncts to this standard:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
265 : 1976	Hydrochloric acid (*third revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
2711 : 1979	Direct reading *p*H meters (*second revision*)
4905 : 1968	Methods for random sampling

3 REQUIREMENTS

3.1 Description

The material shall be in the form of clear white crystals or powder, free from grit, visible impurities and any other foreign matter.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be suitably packed in air tight containers as agreed to between the purchaser and the supplier.

4.1.1 Necessary safeguard shall be provided against risk of dangerous explosions which might occur when the material is heated or subjected to concussion or triturated with organic substances, charcoal, sulphur, sulphides, powdered metallic iron, or other oxidizable substances, and precautions shall be taken at all times to prevent accidents by explosions.

4.2 Marking

Each package shall be legibly and indelibly marked with the following information:

a) Name of the material,
b) Net mass of the material in the container,
c) Indication of the source of manufacture,
d) Date and year of manufacture, and
e) Lot number or batch number in code or otherwise to enable the batch number to be traced from records.

5 SAMPLING

The method of drawing representative samples of the material and criteria for determining the conformity shall be as prescribed in Annex A.

Table 1 Requirements for Potassium Perchlorate for Explosives and Pyrotechnic Industry

(*Clause* 3.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No. of Annex A
(1)	(2)	(3)	(4)
i)	Potassium perchlorate content, percent by mass, *Min*	99·0	A-2
ii)	Moisture content at 105°C, percent by mass, *Max*	0·1	A-3
iii)	Matter insoluble in water, percent by mass, *Max*	0·1	A-4
iv)	Chlorides (as Cl), percent by mass, *Max*	0·1	A-5
v)	Sulphates (as SO_4), percent by mass, *Max*	0·1	A-6
vi)	Chlorates (as $KClO_3$), percent by mass, *Max*	0·05	A-7
vii)	Bromates (as BrO_3), percent by mass, *Max*	0·04	A-8
viii)	*p*H of aqueous extract	5·5 - 7·5	A-9
ix)	Sieving requirements (when in powder form) :		A-10
	a) retained on 125 m IS sieve	Nil	
	b) retained on 90 m IS sieve, percent by mass, *Max*	10·0	

ANNEX A

(*Clauses* 3.2, 5 and *Table* 1)

METHODS OF TEST FOR POTASSIUM PERCHLORATE FOR EXPLOSIVES AND PYROTECHNIC INDUSTRY

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF POTASSIUM PERCHLORATE CONTENT

A-2.1 Weigh accurately about 0·5 g of the sample. Place the sample in a nickel crucible containing chloride free fused sodium chloride. Cover the sample with about 5 g chloride free sodium carbonate. Place the crucible in a hole, bored in asbestos sheet and cover it with a lid so that no reducing gas can enter the crucible. Heat the crucible to dull redness for one hour. Allow it cool and extract the residue with water. Acidify the extract with dilute nitric acid.

A-2.2 Add about 50 ml of 0·1 N $AgNO_3$ to the above extract and boil gently for 20 minutes to coagulate the precipitate. Cool and add 5 ml of 10 percent ferric alum and titrate with 0·1 N ammonium thiocyanate.

A-2.3 Carry out the blank titration following exactly the same procedure as prescribed in **A-2.1** and **A-2.2**.

NOTE — Titre volume of ammonium thiocyanate is equivalent to the chloride and chlorate content, which are in negligible percentage.

A-2.4 Calculation

$$\text{Potassium perchlorate, percent by mass, } Min = \frac{TV \times N \times 0{\cdot}138\,7 \times 100}{M}$$

where

TV = Titre value of the blank;

N = Normality of ammonium thiocyanate solution; and

M = Mass, in g, of the sample taken for the test.

A-3 DETERMINATION OF MOISTURE

A-3.1 Apparatus

A-3.1.1 *Weighing Bottle* — Squat type, with stopper.

A-3.1.2 *Electric Oven* — Capable of being maintained at 105 ± 2°C.

A-3.2 Procedure

Weigh accurately about 5 g of the material into a tared, stoppered, weighing bottle. Keep the bottle in an oven maintained at 105 ± 2°C for 3 hours. Cool in a desiccator and weigh till constant mass.

NOTE — Concentrated sulphuric acid shall not be used as a desiccant.

A-3.3 Calculation

$$\text{Moisture, percent by mass} = 100 \times \frac{M - M_1}{M}$$

where

M = mass, in g, of the sample before drying; and

M_1 = mass, in g, of the sample after drying.

A-4 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-4.1 Apparatus

A-4.1.1 *Filter Crucible* — Gooch or sintered glass (G. No. 4).

A-4.2 Procedure

Weigh accurately about 5 g of the material and dissolve in 200 ml of water. Filter through a tared Gooch or sintered glass crucible (G. No. 4). Wash the residue free of all soluble components and dry to constant mass at 105 ± 2°C.

A-4.3 Calculation

$$\text{Insoluble matter, percent by mass} = 100 \times \frac{m}{M}$$

where

m = mass, in g, of the residue; and

M = mass, in g, of the sample taken for the test.

A-5 DETERMINATION OF CHLORIDES

A-5.1 Reagents

A-5.1.1 *Dilute Nitric Acid* — (1 : 4) (*see* IS 264 : 1976).

A-5.1.2 *Standard Silver Nitrate Solution* — approximately N/20.

A-5.1.3 *Standard Ammonium Thiocyanate Solution* — approximately N/20.

A-5.1.4 *Ferric Alum Indicator* — 10 percent.

A-5.2 Procedure

Weigh accurately about 5 g of the sample and extract in about 100 ml of distilled water. Filter the solution through a Whatman No. 40 filter paper. Wash the residue using about 5 to 10 ml of distilled water and add 20 ml of dilute nitric acid and a measured excess of N/20 silver nitrate solution, usually 20 ml, to the above solution. If a significant palescence due to the precipitation of silver chloride is obtained, shake the flask well till all the silver chloride has coagulated. Titrate with N/20 ammonium thiocyanate using 5 ml of 10 percent ferric alum solution as internal indicator. Carry out a blank titration using all the reagents and following exactly the same procedure as given above.

NOTE — The dilute nitric acid used shall be free from lower oxides of nitrogen which also give a colouration with thiocyanate.

A-5.3 Calculation

$$\text{Chlorides (as Cl), percent by mass, } Max = \frac{(X - Y) \times N \times F \times 0.058\,46 \times 100}{M}$$

where

X = Volume, in ml, of standard ammonium thiocyanate solution required for the blank;

Y = Volume, in ml, of standard ammonium thiocyanate solution required for the sample;

N = Normality of standard ammonium thiocyanate solution;

F = Factor of standard ammonium thiocyanate solution; and

M = Mass, in g, of the sample taken.

A-6 DETERMINATION OF SULPHATES

A-6.1 Apparatus

A-6.1.1 *Nessler Cylinders* — 50-ml capacity.

A-6.2 Reagents

A-6.2.1 *Dilute Hydrochloric Acid* — 1 N.

A-6.2.2 *Barium Chloride Solution* — approximately 10 percent (*m/v*).

A-6.2.3 *Standard Sulphate Solution* — Dissolve 0·148 g of ignited sodium sulphate (Na_2SO_4) in water and dilute to 1 000 ml. One millilitre of this solution contains 0·1 mg of sulphate (SO_4).

A-6.3 Procedure

Dissolve 1 g of the material in 20 ml of water, add 5 ml of dilute hydrochloric acid and 2 ml of barium chloride solution. Transfer to a Nessler cylinder and dilute to mark. Carry out a control test in another Nessler cylinder using 10 ml of standard sulphate solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture.

A-6.3.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that in the control test.

A-7 DETERMINATION OF CHLORATES

A-7.1 Reagents

A-7.1.1 *Aniline Hydrochloride*

Dissolve 18 g of re-distilled aniline in 375 ml of concentrated hydrochloric acid and make up the volume to 500 ml with distilled water, add 2 drops of saturated potassium chlorate solution, shake and allow to stand overnight. Store the reagent in a glass stoppered bottle in the dark.

A-7.1.2 *Standard Potassium Chlorate Solution*

Dissolve 0·2 g of potassium chlorate in water and make up to one litre.

A-7.2 Procedure

Dissolve 5 g of the material in 40 ml of warm water, filter if necessary, and make up to 100 ml in a nessler tube with the aniline hydrochloride agent, compare any blue colouration which develops in 10 minutes with the standard prepared similarly from 5 g of chlorate free potassium perchlorate to which known quantities of standard $KClO_3$ solution has been added.

A-7.3 Calculation

$$\text{Percent chlorate (as } KClO_3 \text{)} = \frac{A \times 0.000\,2 \times 100}{M}$$

where

A = Volume, in ml, of standard potassium chlorate solution added; and

M = Mass, in g, of the sample taken.

NOTE — Commence one series of determinations at the same time since the colour is liable to fade after development. Carry out a preliminary test to ensure that the reagents are effective.

A-8 DETERMINATION OF BROMATES

A-8.1 Reagents

A-8.1.1 *Potassium Iodide Solution* — approximately 10 percent (*m/v*).

A-8.1.2 *Dilute Hydrochloric Acid* — approximately 1 N.

A-8.1.3 *Standard Sodium Thiosulphate Solution* — 0·1 N freshly prepared.

A-8.1.4 *Starch Solution*

Triturate 5 g of pure starch and 0·01 g of mercuric iodide with 30 ml of water in a mortar.

Pour the resulting paste into one litre of boiling water, boil for 3 minutes, allow the solution to cool, and decant off the clear liquid. The solution used for the test shall be freshly prepared.

A-8.1.5 *Potassium Perchlorate* — bromate-free.

A-8.2 Procedure

Weigh accurately about 2 g of the material into a thoroughly cleaned iodine flask and dissolve in about 100 ml of water. Add 5 ml of potassium iodide solution and 5 ml of dilute hydrochloric acid. Stopper the flask and allow to stand in dark for 45 minutes. At the end of this period, add a little of starch solution and titrate the liberated iodine with freshly prepared standard thiosulphate solution.

A-8.2.1 Carry out a blank determination using potassium perchlorate (bromate free) and other reagents used in the test in **A-8.2**.

A-8.3 Calculation

$$\text{Bromate (as } BrO_3 \text{), percent by mass} = 2{\cdot}13 \times \frac{(V_1 - V_2)\, N}{M}$$

where

V_1 = volume, in ml, of standard sodium thiosulphate solution required for the test with the material;

V_2 = volume, in ml, of standard sodium thiosulphate solution required for the blank determination;

N = normality of standard sodium thiosulphate solution; and

M = mass, in g, of the material taken for the test.

A-9 DETERMINATION OF *p*H

A-9.1 Apparatus

A-9.1.1 p*H Meter*

Use a standard laboratory *p*H meter (*see* IS 2711 : 1979).

A-9.2 Procedure

Take about 5 g of the material and dissolve it in 100 ml of neutral distilled water. Allow to stand for 5 minutes and then measure the *p*H of the solution, using a standard *p*H meter.

A-9.2.1 The *p*H value of the solution shall be within the limits specified in Table 1.

A-10 DETERMINATION OF SIEVING REQUIREMENT

A-10.1 Procedure

Sieve the material through 125 m and 90 m IS sieves respectively using a light camel hair brush. Weigh accurately the material retained on the sieve and report as percent by mass.

ANNEX B

(*Clause* 5)

SAMPLING OF POTASSIUM PERCHLORATE FOR EXPLOSIVES AND PYROTECHNIC INDUSTRY

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing samples, the following precautions and directions shall be observed.

B-1.1 Necessary safeguards as prescribed in **4.1.1** shall be provided in taking out the material from the containers for sampling.

B-1.2 The samples and the sampling implement shall be well protected from any possible contamination.

B-1-3 The sampled material shall be placed in a suitable, dry and clean container which shall be marked with all the relevant details after filling.

B-1.4 Samples shall be taken from different parts of the selected containers with the help of suitable sampling implements.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers of potassium perchlorate manufactured at a time and under similar conditions of production shall be grouped together to constitute a lot. Each lot shall be tested separately for testing the various requirements of the specification.

B-2.2 The number of containers to be selected for the sample shall be as given in Table 2.

These containers shall be selected at random from the lot as per IS 4905 : 1968.

B-2.3 Preparation of Test Samples

From each of the selected containers a representative portion of the material shall be taken, which shall be sufficient for carrying out tests for all characteristics given in the specification.

Table 2 Scale of Sampling

(*Clause* B-2.2)

No. of Containers in a Lot (1)	No. of Containers to be Selected (2)
Up to 25	3
26 to 50	4
51 to 100	5
101 and above	7

B-2.3.1 *Composite Sample*

Out of these portions, a small but equal quantity of material shall be taken and mixed thoroughly to make a composite sample.

B-2.3.2 *Individual Sample*

The remaining portion of the material from each selected container shall constitute an individual test sample.

B-2.4 Number of Tests

Tests for description and potassium perchlorate content shall be conducted on the individual sample whereas tests for all other characteristics shall be conducted on the composite sample.

B-2.5 Criteria for Conformity

The lot shall be deemed to conform to the specifications if all the test results according to **B-2.4** satisfy the corresponding requirements.

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